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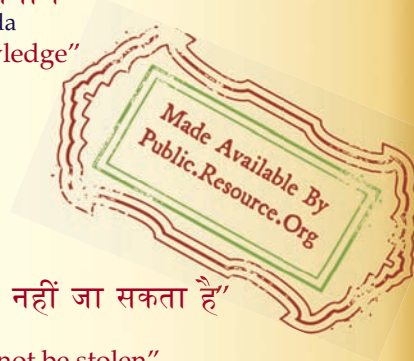
IS 228-20 (2003): Methods for Chemical Analysis of Steels, Part 20: Determination of Carbon and Sulphur by Infra Red Absorption Method (for Carbon 0.005 to 2 Percent and Sulphur 0.001 to 0.35 Percent) [MTD 4: Wrought Steel Products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

इस्पात के रासायनिक विश्लेषण की प्रणाली

भाग 20 अवरक्त अवशोषण द्वारा इस्पात में कार्बन तथा सल्फर का निर्धारण
(कार्बन 0.005 प्रतिशत से 2 प्रतिशत तथा सल्फर 0.001 प्रतिशत से 0.35 प्रतिशत)

(दूसरा पुनरीक्षण)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 20 DETERMINATION OF CARBON AND SULPHUR BY INFRA RED
ABSORPTION METHOD (FOR CARBON 0.005 TO 2 PERCENT AND
SULPHUR 0.001 TO 0.35 PERCENT)

(*Second Revision*)

ICS 77.080.20

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FOREWORD

This Indian Standard (Part 20) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 228, which was first published in 1952 and subsequently revised in 1959, covered the chemical analysis of plain carbon and low alloy steels, along with pig iron and cast iron. It was revised again to make it comprehensive in respect of steel analysis and to exclude pig iron and cast iron which were being covered in separate standards. During its second revision the standard has been split up in several parts.

This part covers the method for determination of carbon and sulphur. The other parts of this series are:

- (Part 1) : 1987 Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent) (*third revision*)
- (Part 2) : 1987 Determination of manganese in plain-carbon and low alloy steels by arsenite method (*third revision*)
- (Part 3) : 1987 Determination of phosphorus by alkalimetric method (*third revision*)
- (Part 4) : 1987 Determination of total carbon by gravimetric method (for carbon greater than or equal to 0.1 percent) (*third revision*)
- (Part 5) : 1987 Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel greater than or equal to 0.1 percent) (*third revision*)
- (Part 6) : 1987 Determination of chromium by persulphate oxidation method (for chromium ≥ 0.1 percent) (*third revision*)
- (Part 7) : 1990 Determination of molybdenum by alpha-bezoinoxime method (for molybdenum 1 percent and not containing tungsten) (*third revision*)
- (Part 8) : 1989 Determination of silicon by gravimetric method (for silicon 0.05 to 0.50 percent) (*third revision*)
- (Part 9) : 1989 Determination of sulphur in plain carbon steels by evolution method (for sulphur 0.01 to 0.25 percent) (*third revision*)
- (Part 10) : 1989 Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum 0.01 to 1.5 percent) (*third revision*)
- (Part 11) : 1990 Determination of total silicon by reduced molybdosilicate spectro photo metric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent) (*third revision*)
- (Part 12) : 2001 Determination of manganese by periodate spectrophotometric method in low and high alloy steels (for manganese 0.01 to 5.0 percent) (*fourth revision*)
- (Part 13) : 1982 Determination of arsenic
- (Part 14) : 1988 Determination of carbon by thermal conductivity method (for carbon 0.005 to 2.000 percent)
- (Part 15) : 1992 Determination of copper by thiosulphate iodide method (for copper 0.05 to 5 percent) (*second revision*)
- (Part 16) : 1992 Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent) (*second revision*)

(Continued on third cover)

*Indian Standard***METHODS FOR CHEMICAL ANALYSIS OF STEELS****PART 20 DETERMINATION OF CARBON AND SULPHUR BY INFRA RED
ABSORPTION METHOD (FOR CARBON 0.005 TO 2 PERCENT AND
SULPHUR 0.001 TO 0.35 PERCENT)***(Second Revision)***1 SCOPE**

This standard (Part 20) describes the method for determination of carbon in the range from 0.005 to 2 percent and sulphur from 0.001 to 0.35 percent in steel.

2 SAMPLING

The samples shall be drawn and prepared as prescribed in the relevant Indian Standards.

3 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents shall be employed for the test.

4 DETERMINATION OF CARBON AND SULPHUR**4.1 Outline of the Method**

The sample is burnt with accelerator at high temperature in a flow of oxygen in a high frequency induction furnace. The oxygen flow serves both as oxidizing agent and as system carrier gas. Carbon in the sample is converted to carbon dioxide and sulphur to sulphur dioxide. These are carried by oxygen stream into the non-dispersive infra red absorption analyzer where these gases are measured quantitatively.

4.2 Reagents

4.2.1 Oxygen — Purity 99.9 percent (purified by passing through magnesium perchlorate and soda asbestos).

4.2.2 Magnesium Perchlorate, particle size less than 1.5 mm.

4.2.3 Absorbants — Soda asbestos or ascarite.

4.2.4 Tungsten, copper or tin with low carbon and sulphur (below 0.000 5 percent).

NOTE — Tungsten granules are preferred over others.

4.2.5 Trap Materials — Ascarite for removing traces of carbon dioxide and molecular sieve No. 5A for removing moisture.

4.2.6 Standard Samples — The standard samples of steels of known carbon and sulphur, preferably in the same range as the samples, are to be used for calibration.

4.3 Apparatus

An automatic carbon and sulphur analyzer consists of a high frequency induction furnace precision flow regulator and tandem IR detectors as shown in the schematic diagram (see Fig. 1). Oxygen gas of commercial purity is purified through a series of filters and passed into induction furnace. This oxygen stream carries the evolved gases, namely, carbon dioxide and sulphur dioxide due to overcombustion of steel sample.

After passing through the moisture and dust filters, the gas is passed through precision flow regulator and the regulated gas is conveyed to non dispersive infra red detector arranged in tandem, one for carbon dioxide and another for sulphur dioxide measurement. Some of such units may have facilities for recording the sample weight and display of blank weight-corrected results in terms of percentage or ppm of carbon and sulphur. Follow the detailed instruction manuals for the operation of the instrument.

4.3.1 High precision balance for weighing 0 to 100 g with an accuracy of 0.1 mg.

4.3.2 Crucibles — Ceramic crucibles of dimensions suitable for the furnace which can withstand thermal shock and high temperature and having low consistent carbon and sulphur contents.

5 PROCEDURE**5.1 Equipment Preparation**

Turn on the instrument and allow sufficient time to stabilize the system. Change dust filters, traps, etc, as required. Clean the furnace chamber and test the furnace and analyzer for absence of leaks. Fix the parameters like analysis time, sample identity, etc.

5.2 Crucible Preparation

Preheat the ceramic crucibles in a flow of oxygen in a furnace kept at 1 000°C for about 2^h. Cool and remove these crucibles and store them in a desiccator.

5.3 Sample Preparation

It is preferable to have all samples and standards in the form of small blocks, chips or wires. These have to be filed to remove surface contamination, washed in acetone and dried.

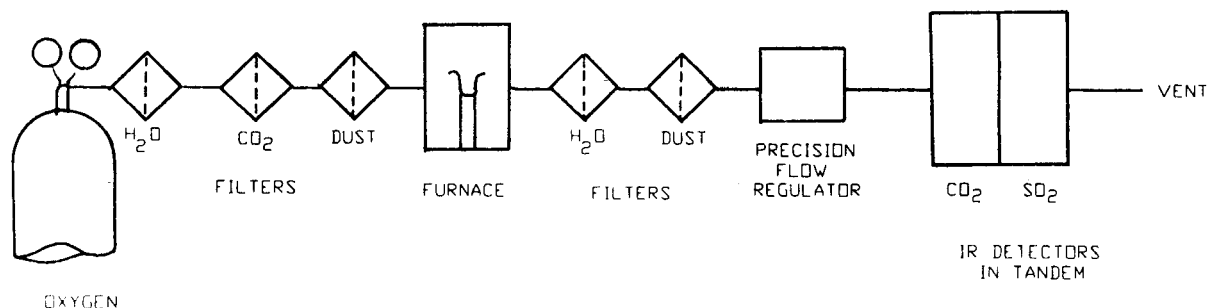


FIG. 1 AUTOMATIC CARBON AND SULPHUR ANALYZER

5.4 Blank Determination

Transfer about 1g of tungsten or other accelerator granules into the crucible. Load the crucibles in the induction furnace and run the analysis cycle. The output of the two IR detectors are converted to percentage of carbon and sulphur respectively and used for blank correction. If provision exists, enter these values into the memory for automatic blank corrections during further analysis. Repeat the blank run in order to ensure good precision.

5.5 Calibration

Weigh about 1 g of sample prepared as per 5.3 and transfer into the crucible. Add 1 g of accelerator to the

crucible and insert it into the furnace. Run through the analysis cycle. Adjust the output of the respective IR detectors to the standard values after deducting blanks for carbon and sulphur. Repeat the calibration procedure with the same standard and proceed further only, if consistent results are obtained. If multi point calibration facility is available in the instrument, run one low and one high standard following the above procedure.

5.6 Analysis of Sample

Run the sample as per the procedure given in 5.5. Apply corrections for blank, if it is not done automatically by the instrument. Repeat the analysis to check the consistency of the results.

(Continued from second cover)

- (Part 17) : 1998 Determination of nitrogen by thermal conductivity method (for nitrogen up to 0.4 percent)
(*second revision*)
- (Part 18) : 1998 Determination of oxygen by instrumental method (*second revision*)
- (Part 19) : 1998 Determination of nitrogen by steam distillation (*second revision*)
- (Part 21) : 2003 Determination of copper by spectrophotometric method (for copper 0.02 to 0.50 percent)
(*second revision*)
- (Part 22) : 2003 Determination of total hydrogen in steel by thermal conductivity method (hydrogen 0.1 to 50 ppm)
- (Part 23) : 2003 Determination of nitrogen in steel by optical emission spectrometer (nitrogen 0.002 to 1.0 percent)
- (Part 24) : 2003 Determination of nitrogen in steel by inert gas fusion — Thermal conductivity method (nitrogen 0.001 to 0.2 Percent)

In this revision the Infra red absorption method after combustion in induction furnace has been prescribed for determination of carbon and sulphur in steel. Assistance has been derived from ISO 4935:1989 'Steel and iron — Determination of sulphur content — Infra red absorption method after combustion in an induction furnace' and ISO 9556:1989 'Steel and iron — Determination of total carbon content — Infra red absorption method after combustion in an induction furnace' for preparation of this Indian Standard.

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated is to be rounded off it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

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This Indian Standard has been developed from Doc: No. MTD 2 (3444).

Amendments Issued Since Publication

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